USERS GUIDE

Deep UV Excitation Spectrophotometer

...Spectra\10692G\DUVEx User Guide.doc

McPHERSON

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General Description

The McPherson Vacuum UV Spectrophotometer is for diverse spectral measurements across a broad wavelength range, extending from the vacuum ultraviolet to the visible. The overall range may be expanded with addition of alternate sources, detectors and gratings.

The instrument is equipped with a vacuum spectrometer for short wavelength excitation. It is mounted contiguous to the vacuum tight sample chamber. The emission spectrometer works at vacuum or atmosphere (optionally purged with inert gas.) The excitation and emission instruments can be scanned synchronously. Alternatively, one can be positioned at zero order or at the desired excitation/emission wavelength, etc. Scanning both instruments individually or together and with an offset to reject fluorescence is also possible.

There is one principle sample location, equipped with a multiple sample holder. The sample location is suitable for various unique measurement modes. For example, it may be used for reflectance and fluorescence or luminescence, etc. By moving the detector to the alternate sample chamber exit port one can measure transmission or absorbance. Samples in either position can be indexed into the light beam and selected under vacuum. The chamber is equipped with a removable and vacuum compatible Si detector.

The sample chamber includes a port for window location. Insertion of an optional small diverting mirror allows introduction of other (laser, x-ray) sources without disrupting the principle setup.

The windowless deep VUV source can fill the system with wavelengths to at least 20nm while two turbo pumps maintain high vacuum.

The optical mechanical assembly is built on a supporting table structure for ease of transport and set up. The computer may be located on a separate table. All cables and lines are labeled for easy set up.

The McPherson Vacuum UV Spectrophotometer is intended for use in university or research laboratories. It attempts to provide a well-working core system for investigation. It allows users flexibility to select measurement modes, as well as possibilities to expand, or add to the system in the future.

Refer to unique Instruction Manuals for the system elements in addition to this general guide.

Vacuum Spectrophotometer, Top View

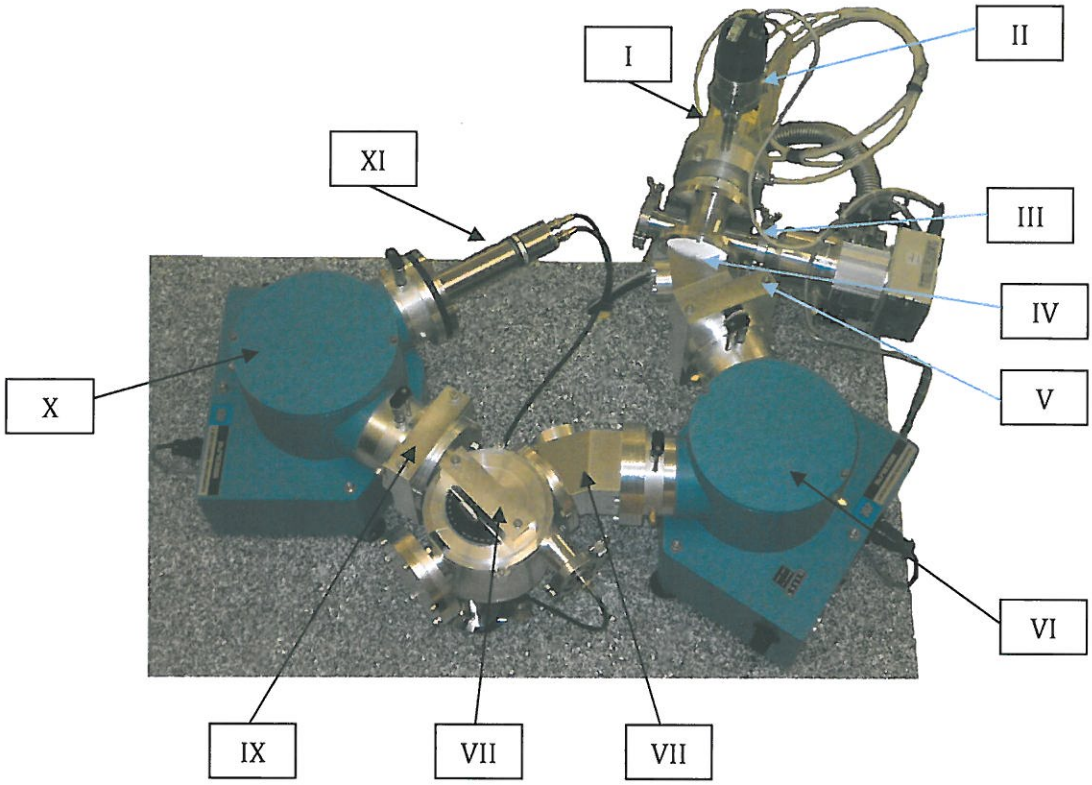
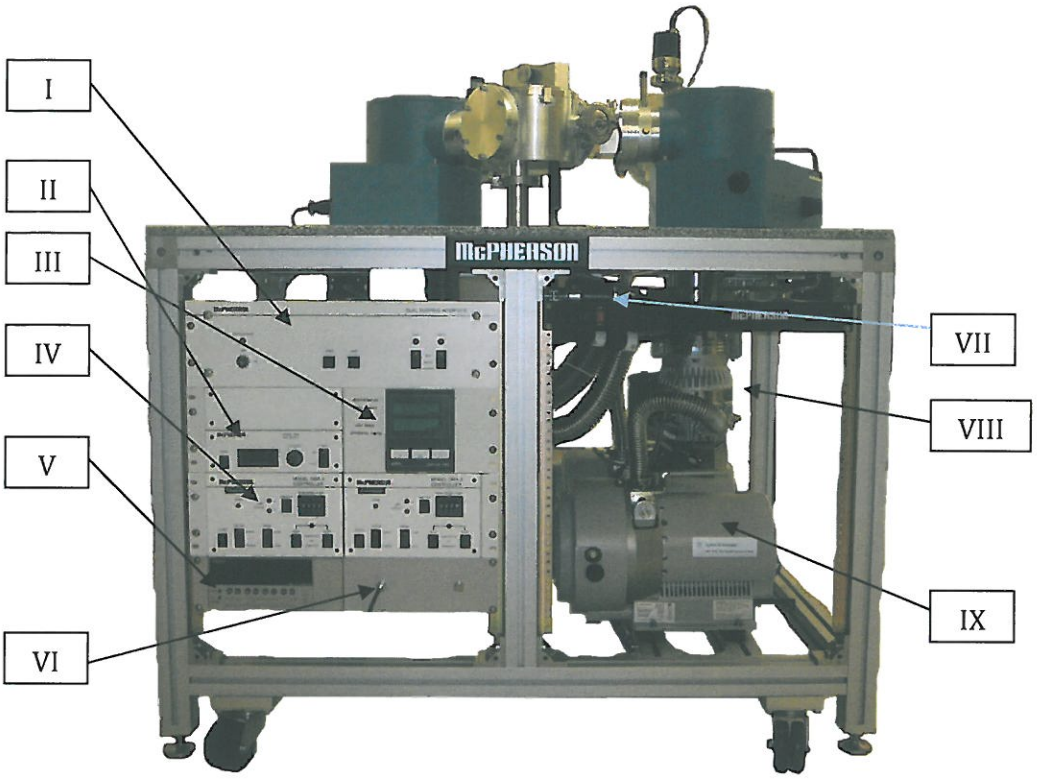


Table top assembly holds:

I. Hollow cathode Model 629 source with gas inlet and water cooling hoses
II. Vacuum gage to control gas pressure in the Hollow cathode lamp
III. Differential pumping unit with turbo pump
IV. Reflective condenser
V. Excitation wavelength sorting filter wheel
VI. Excitation monochromator Model 234/302 (I) with stepper motor drive
VII. Reflective condenser
VIII. Sample chamber with multiple sample mounting lid, focusing mirror and Si detector
IX. Emission wavelength sorting filter holder
X. Emission monochromator Model 234/302 (II) with stepper motor drive
XI. PMT housing

Units listed have separate instruction manuals provided as paper copy or on CD

Vacuum Spectrophotometer, Front View



I. Pump controller. Controls both pumps
II. PMT power supply
III. Vacuum gauge controller. Top display for vacuum gauge on the sample chamber, bottom – light source
IV. Controllers for monochromators Model 234/302 (I) and (II)
V. Keithley picoammeter
VI. Interface for PMT and Si detector signals acquisition
VII. Sample chamber vacuum gauge
VIII. Turbo pump
IX. Primary pumps

Units listed have separate instruction manuals provided as paper copy or on CD

Nitrogen Vent and Water Connections

Nitrogen:
For best system life, faster pumping times, and cleanliness we strongly recommend a source of dry Nitrogen gas is available to "back fill" the vacuum system. When the vacuum is interrupted, the instrument volume fills with dry-Nitrogen rather than room air (eliminating humidity or contaminants, etc.) Have a tank of dry Nitrogen gas, with regulator, and suitable length of clean (new) tubing and hose connection fittings ready at installation time. The connector to connect dry Nitrogen is at the back of the table. It is ¼ Swagelok connector.

Water
The Hollow cathode lamp requires flowing water (chilling is not required). Inlet and outlet lines are indicated on the lamp housing.

Compressed Air:
Not required with this system.
Only systems with a gate valve (or valves) require compressed air. Air should be clean and free of oil and water as is typical for most laboratory 'house air.' The supply must have at least 50psi pressure (75 – 100 preferred.)

Coating Maintenance

The mirrors in the Vacuum Spectrophotometer are ultraviolet-enhanced multilayer reflective coatings, Al + Mg F2.

CAUTION: This is a fragile, soft, first surface coating.

DO NOT wipe the surface. The possibility to damage the coating (adding sleeks) is high. Wiping the optics should only be attempted in dire circumstances.

The best maintenance is to keep the optics safe from dust or contact of any kind. Keep the mirrors covered or stored under cover, to prevent build up of dust, etc.

The optics can be blown free of particulates using (ionized) dry-Nitrogen gas or clean, dry compressed air if available.

Operating the Instrument

Do not initiate pumping yet!

Before starting pumping the gas supply tube of the Hollow cathode lamp has to be plugged or closed with suitable valve. A needle valve is required to operate Hollow cathode lamp properly. The connectors on the gas and water tubes are ¼ inch Swagelok connectors.

Install gratings in both monochromators.

Connect water supply to the Hollow cathode lamp. Turn the water on and check for any leaks. If there are no leaks turn electronics on by turning the key in the pump controller.

To start pumps push "PUMP" button on the pump controller. Both pumps start simultaneously.

When the vacuum level is below 1*10E-4 turn the Hollow cathode lamp on. The vacuum level of the system gets lower and the pumps work harder. But once the plasma is ignited and minimum gas flow to keep plasma burning is found the vacuum level improves.

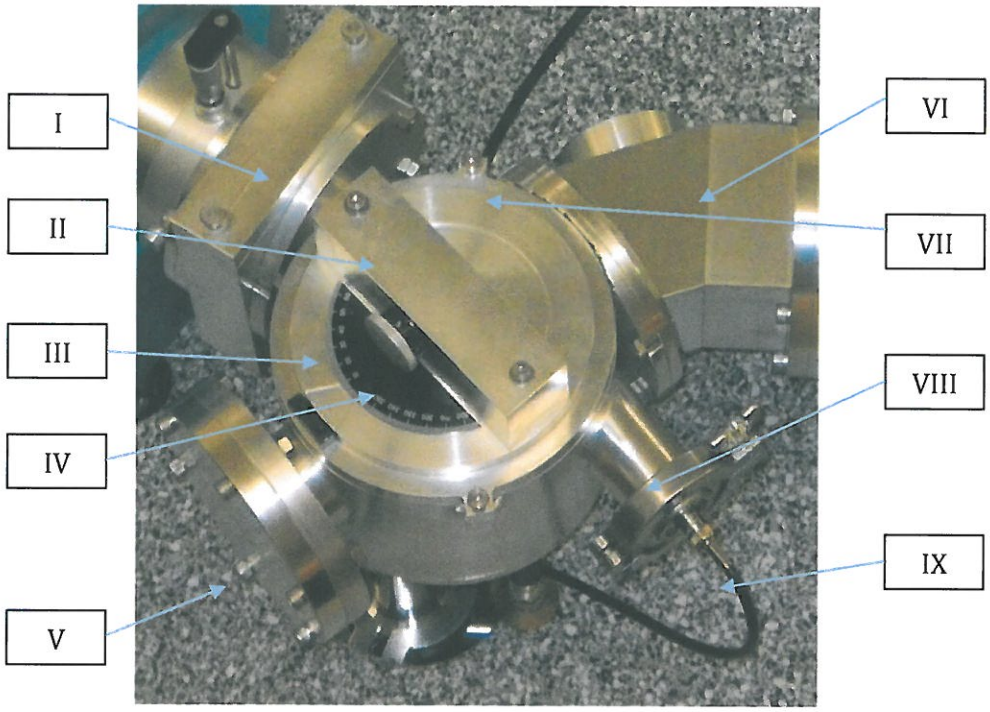
Sample Chamber

The sample chamber (see picture below) lid is fastened down via two (2) shoulder screws on its peripheral edge. These screws are held in tight position in external blocks that guide the cover into place. Loosening these screws enables the lid to be lifted.

Inside you see the focusing mirror in its mount. It receives light from the excited sample and focuses it on the entrance slit of the emission monochromator. The sample mounting assembly is normally located in the sample-wheel protruding through the top cover of the chamber. For alignment purposes or transmission measurements a target can be simply positioned on the inside base of the sample chamber. Alternately, with sample wheel in position a frosted glass or similar sample allows to view sample illumination via the transmission port when covering flange is removed.

The Si detector is mounted directly behind the sample holder allowing a measurement of all the light that reaches the sample position.

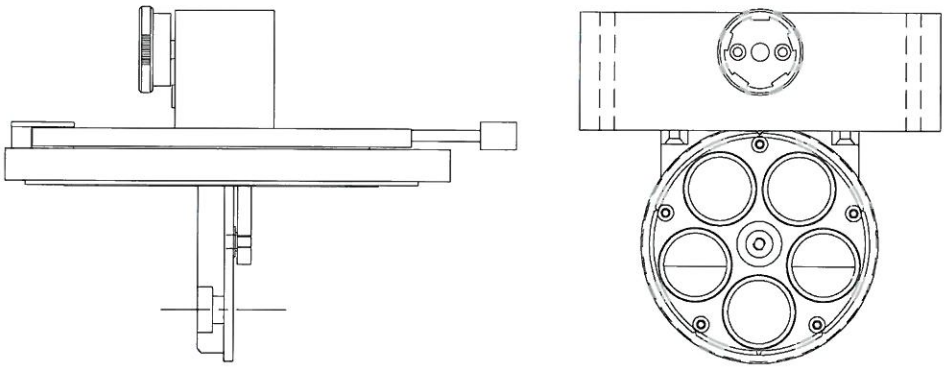
Exterior view of sample chamber



I. Filter holder
II. Sample wheel cover
III. Sample angle scale
IV. Sample rotation and indication knob
V. Transmission/View port
VI. Reflective condenser
VII. Top cover shoulder screw
VIII. Laser input port
IX. Si detector electrical feedthrough

Sample and Location

Samples are accommodated by placement in the reflectance or transmission turret inserted through elongated port, through the sample chamber cover.



Five position sample wheel seen protruding through sample chamber top cover

Samples mounted in the reflectance / emission five position wheel through elongated slots in the top cover also have the possibility to be adjusted with respect to angle. This allows rejecting direct reflection of excitation energy and preferential collection of luminescence, etc. The angular position is set via extended lever; angle position is indicated on the scale.

Sample Alignment Procedure (in air) with Zero Order

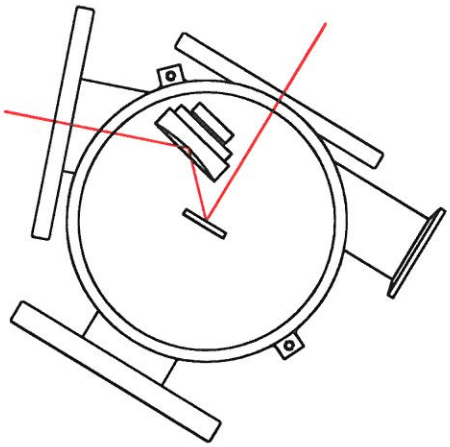
Use a Mercury lamp for alignment. Put the Mercury lamp instead of the Hollow cathode lamp.

1. Maximize zero order at the exit slit of the excitation monochromator
2. Maximize zero order onto center of sample (use frosted glass target in wheel)
3. Maximize zero order onto emission monochromator entrance slit, be sure to be well centered, then
4. Insert sample holder wheel
5. Insert sample into sample holder, and position sample to be illuminated with zero order light (as was observed in point 3.)
6. Observe emitted light or scattered zero order light on exit slit of emission monochromator

Note: when first installing your sample if the alignment at the emission monochromator entrance slit has changed first slightly rotate the sample.

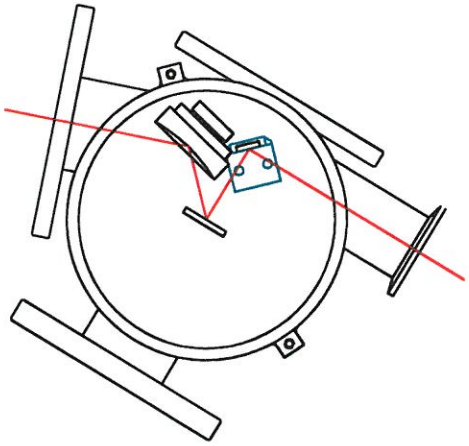
Each monochromator is equipped with a "HOME" position switch. The switch provides quick and accurate recalibration if the wavelength count is lost in the instrument control software. This switch is intended only for use in a software control environment. The "Home" function is disabled in "Local" mode. The value of the "Home" switch is indicated on the Certificate of Performance. For more information refer to 789A-3 controller and Software manuals.

Procedures with Open Sample Chamber



Before scanning any sample, proper alignment should be confirmed. With the excitation monochromator positioned at Zero Order (on counter 00000) a mirror placed into the sample-space can be rotated until the zero order light beam reaches the focusing mirror and subsequently the entrance slit of the emission spectrometer.

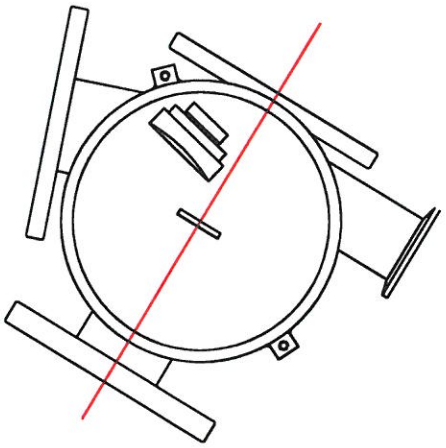
The reflected light spot from the mirror should be centered on the collimating mirror. Beam trajectory adjustment should be accomplished via sample angle. The collection mirror is pre set and does not require adjustment.



For REFLECTING and EMITTING samples

After confirmation of beam trajectory, one can insert samples in the holder. Samples have scatter when exposed to the excitation beam. This scatter is sufficient to direct and see it on the entrance slit of the emission monochromator. Direction can be found by viewing the spot through the laser entrance / accessory port or by photometric "peaking" of signal at zero order.

The small laser diverting mirror can be installed on the dowel pin located mount when introducing laser excitation.



For TRANSMISSION samples

To measure transmission, the sample should be placed in the sample position and adjusted as described under "scanning and analysis" be sure that the raw beam (zero order light from the 234/302 excitation monochromator) reaches the detector or the entrance slit of the emission monochromator. These items must be moved to the transmission port for this measurement. The cover-mounted 5-position sample turret can be used for holding transmission samples.

Nominal Specifications

Light Source
Hollow cathode lamp
Excitation Wavelength output from ~20nm

Light Source Condenser, Model 615
Source condenser with concave spherical reflector at 70 degree AOI

Excitation/Emission Spectrometer, Model 234/302
Focal length 200mm (nominal)
Aperture f/4.5
Grating 1200g/mm concave spherical, 140nm blaze (4nm/mm dispersion), Al/MgF2 coated
Grating 2400g/mm concave spherical, 80-130nm blaze (2nm/mm dispersion), Platinum coated
Adjustable entrance and exit slits
Stepper driven wavelength scanning from software
Manual operation override
O-ring sealed vacuum to 10E-6 torr range

Vacuum Sample Chamber
Adjustable focusing and collection optics
Coating VUV, Aluminum with Magnesium Fluoride overcoat
Transmission Position / Sample viewing window
Sample holder accepting up to five 25mm diameter samples
Samples may be selected under vacuum
Reflection / Emission position allows angular tilt (min ±10deg)
Accessory port(s) for direct illumination of sample from additional (laser, x-ray sources)
O-ring sealed vacuum to 10E-6 torr range

Detector (one exit port)
Photomultiplier housing
+ R6095 PMT for 300 to 650nm
Si diode detector

Software
McPherson Spectrometer Control Software

For additional information please refer to unique Instruction Manuals for the system elements.